SINTESIS, KARAKTERISASI DAN AKTIVITAS KATALIS $Mg_{1-x}Zn_xF_{0,66}(OH)_{1,34}$ PADA REAKSI TRIMETILHIDROKUINON DAN ISOFITOL

Oleh :

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1412100013

Dosen Pembimbing :

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PENDAHULUAN

Katalis

PENDAHULUAN

PENDAHULUAN

Katalis

Katalis Homogen

H_2SO_4, HCl, BF_3, $AlCl_3$ dan $FeCl_2$/HCl
(Bonrath & Netscher, 2005)

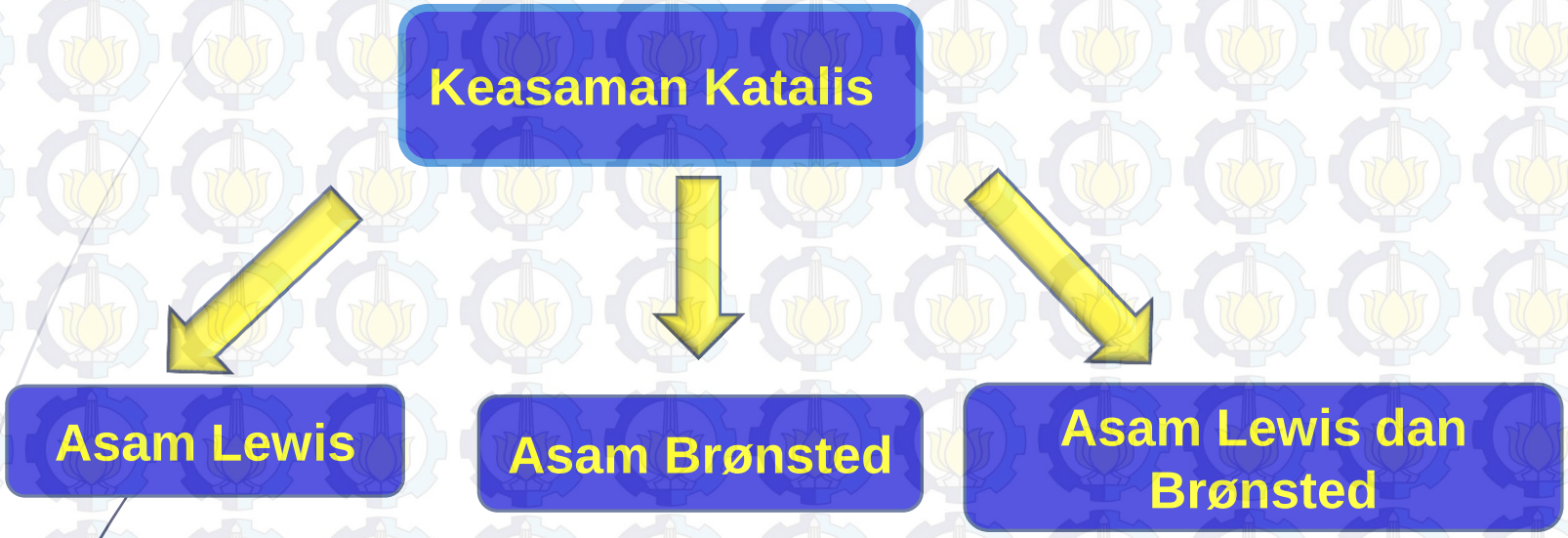
- Sulit dipisahkan
- Korosif
- Katalis hanya bisa dipakai sekali (Lien, 2012)

Katalis Heterogen

Nafion/SiO_2 (Laufer dkk., 2005), $M(OTf)_3$ (Schrager & Bonrath, 2007)

- Mudah dipisahkan
- Tidak Korosif
- Katalis bisa dipakai kembali (Nguyen dkk., 2012)

PENDAHULUAN



RUMUSAN MASALAH

Katalis MgF_2 → Keasaman <<

Modifikasi Keasaman

- Brønsted → Penambahan OH
- Lewis → Doping Zn

$Mg_{1-x}Zn_xF_{0,66}(OH)_{1,34}$

- Keasaman
- Konversi
- *Yield*

TUJUAN PENELITIAN

$MgF_{0,66}(OH)_{1,34}$ + Zn

$Mg_{1-x}Zn_xF_{0,66}(OH)_{1,34}$ *

* X = 0 ; 0,025 ; 0,05 ; 0,075 ; 0,1 ; 0,15

Aktivitas

TMHQ + Isofitol

Manfaat

Aplikasi pada reaksi TMHQ dan Isofitol

2 METODOLOGI PENELITIAN

Sintesis Katalis

$Mg_{1-x}Zn_xF_{0,66}(OH)_{1,34}$ *

Karakterisasi

- XRD
- FTIR
- Adsorpsi Piridin-FTIR
- Adsorpsi gas N_2

Uji Katalisis

TMHQ + Isofitol

↓

Produk

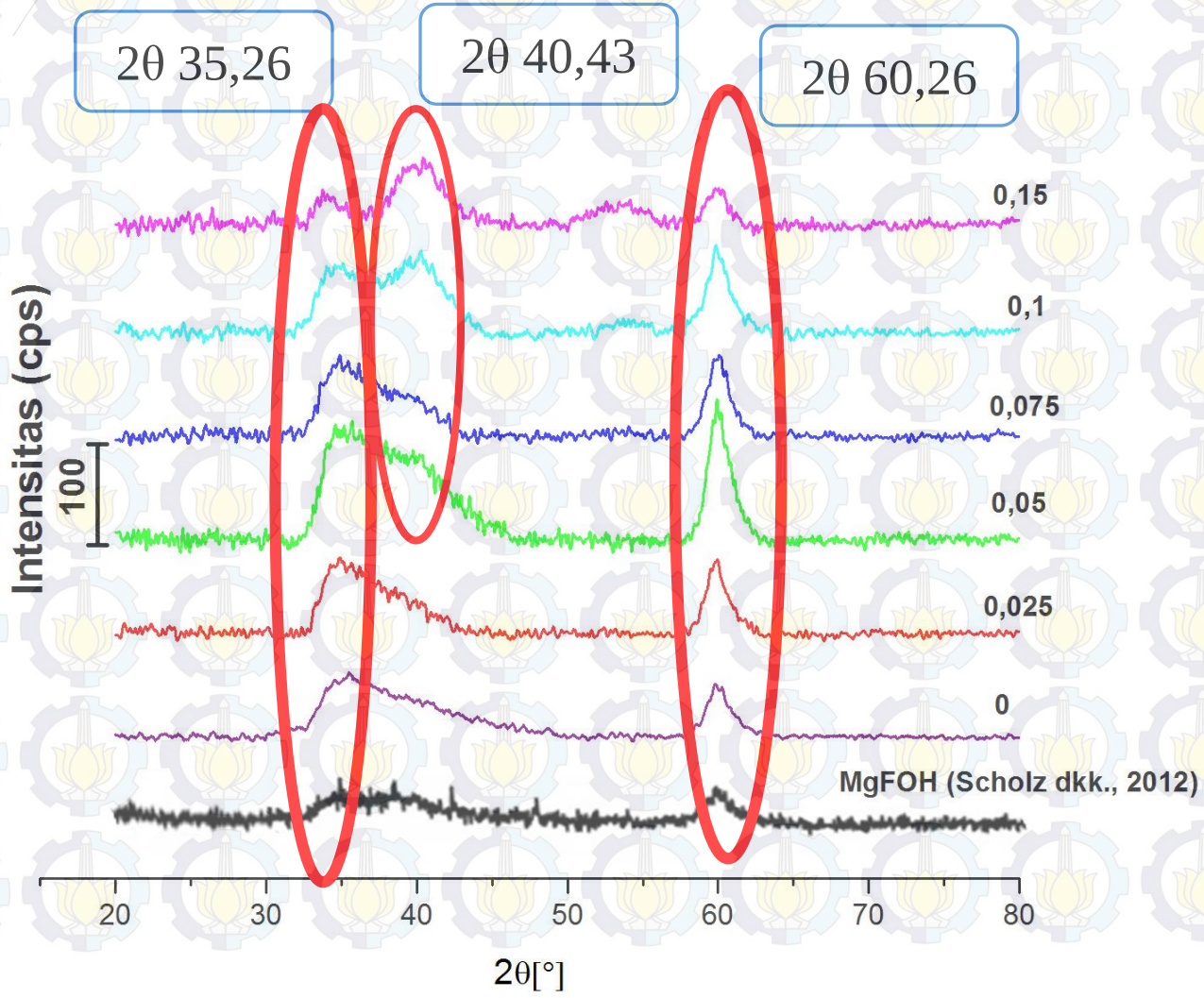
↓

UV-Vis

*x = 0 ; 0,025 ; 0,05 ; 0,075 ; 0,10 dan 0,15

3 HASIL DAN PEMBAHASAN

Difraktogram $Mg_{1-x}Zn_xF_{0,66}(OH)_{1,34}$



3 HASIL DAN PEMBAHASAN

Tabel pergeseran 2θ pada katalis $Mg_{1-x}Zn_xF_{0,66}(OH)_{1,34}$

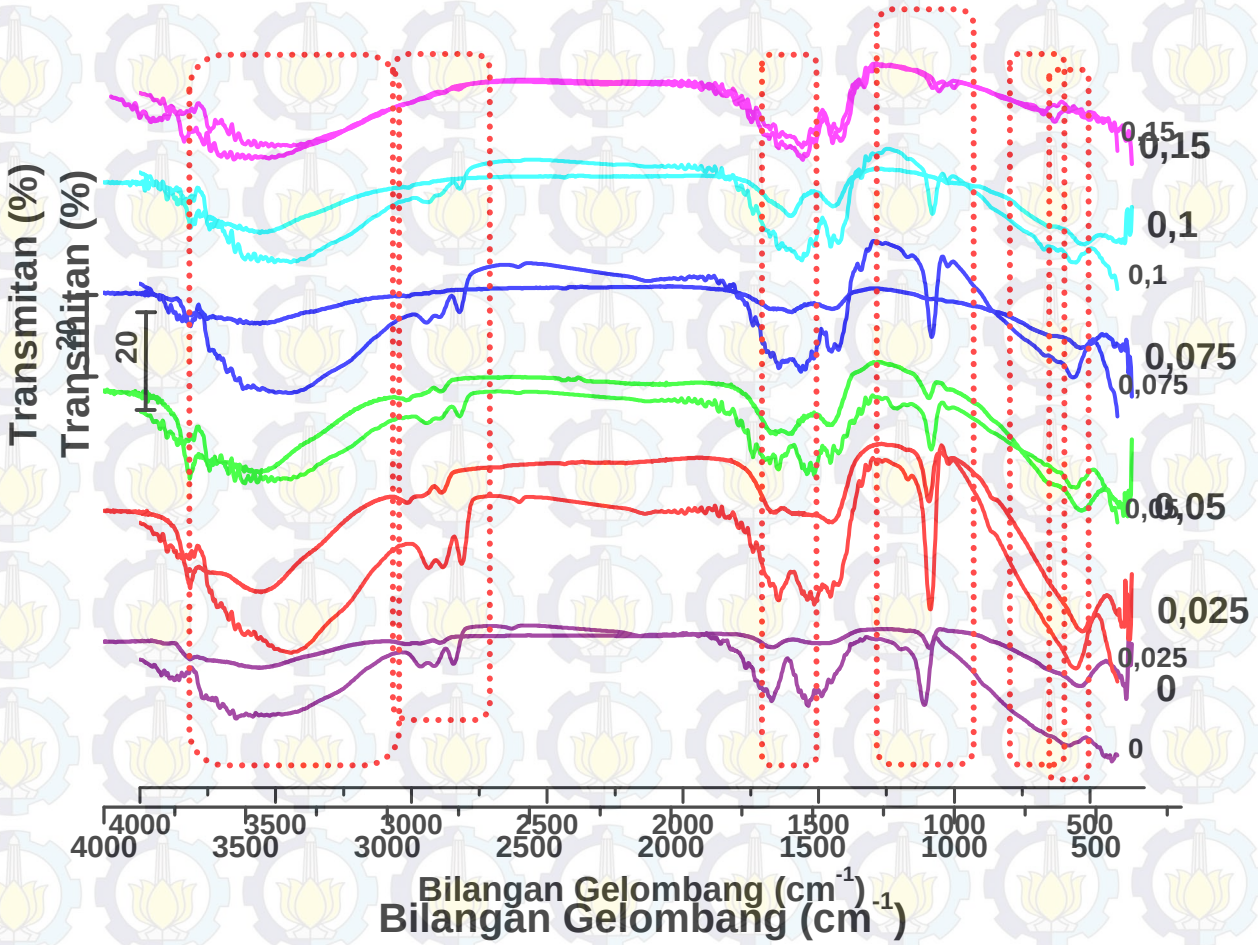
Nilai x pada katalis $Mg_{1-x}Zn_xF_{0,66}(OH)_{1,34}$	Puncak 1		Puncak 2		Puncak 3	
	2θ(°)	Int. (cps)	2θ(°)	Int. (cps)	2θ(°)	Int. (cps)
0	35,26	141	-	-	60,26	108
0,025	34,83	181	-	-	59,99	146
0,05	35,63	273	39,97	259	60,21	268
0,075	35,29	209	40,06	172	59,99	167
0,1	34,01	246	39,44	238	60,06	193
0,15	34,26	202	39,21	225	60,24	142

3 HASIL DAN PEMBAHASAN

Sebelum Kalsinasi

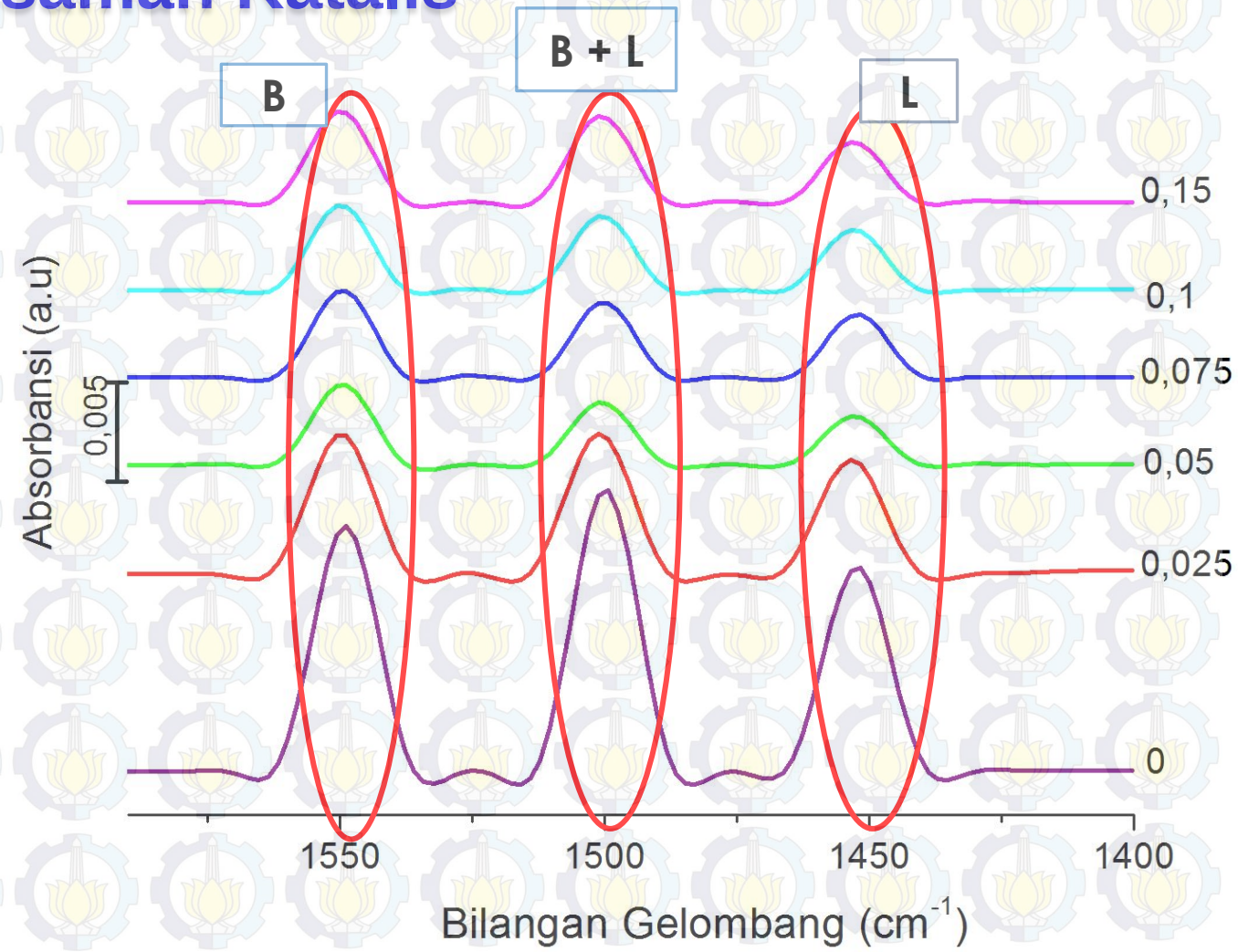
Setelah Kalsinasi

FTIR



HASIL DAN PEMBAHASAN

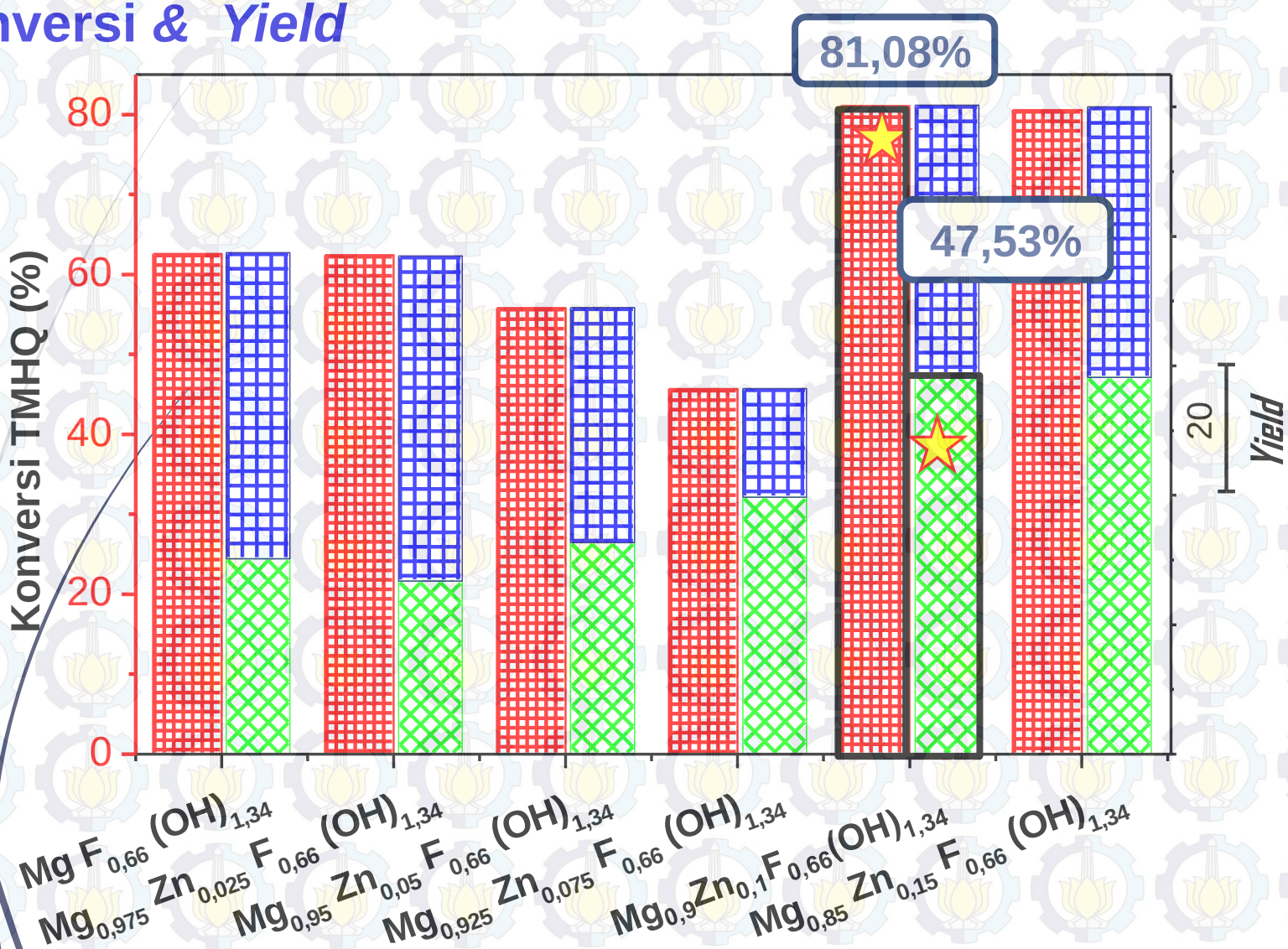
Profil Keasaman Katalis



Luas Permukaan Katalis

Katalis	$S_{(BET)}$ (m^2/g)
$MgF_{0,66}(OH)_{1,34}$	26,884
$Mg_{0,975}Zn_{0,025}F_{0,66}(OH)_{1,34}$	202,757
$Mg_{0,95}Zn_{0,05}F_{0,66}(OH)_{1,34}$	369,603
$Mg_{0,925}Zn_{0,075}F_{0,66}(OH)_{1,34}$	308,079
$Mg_{0,9}Zn_{0,1}F_{0,66}(OH)_{1,34}$	253,720
$Mg_{0,85}Zn_{0,15}F_{0,66}(OH)_{1,34}$	236,325

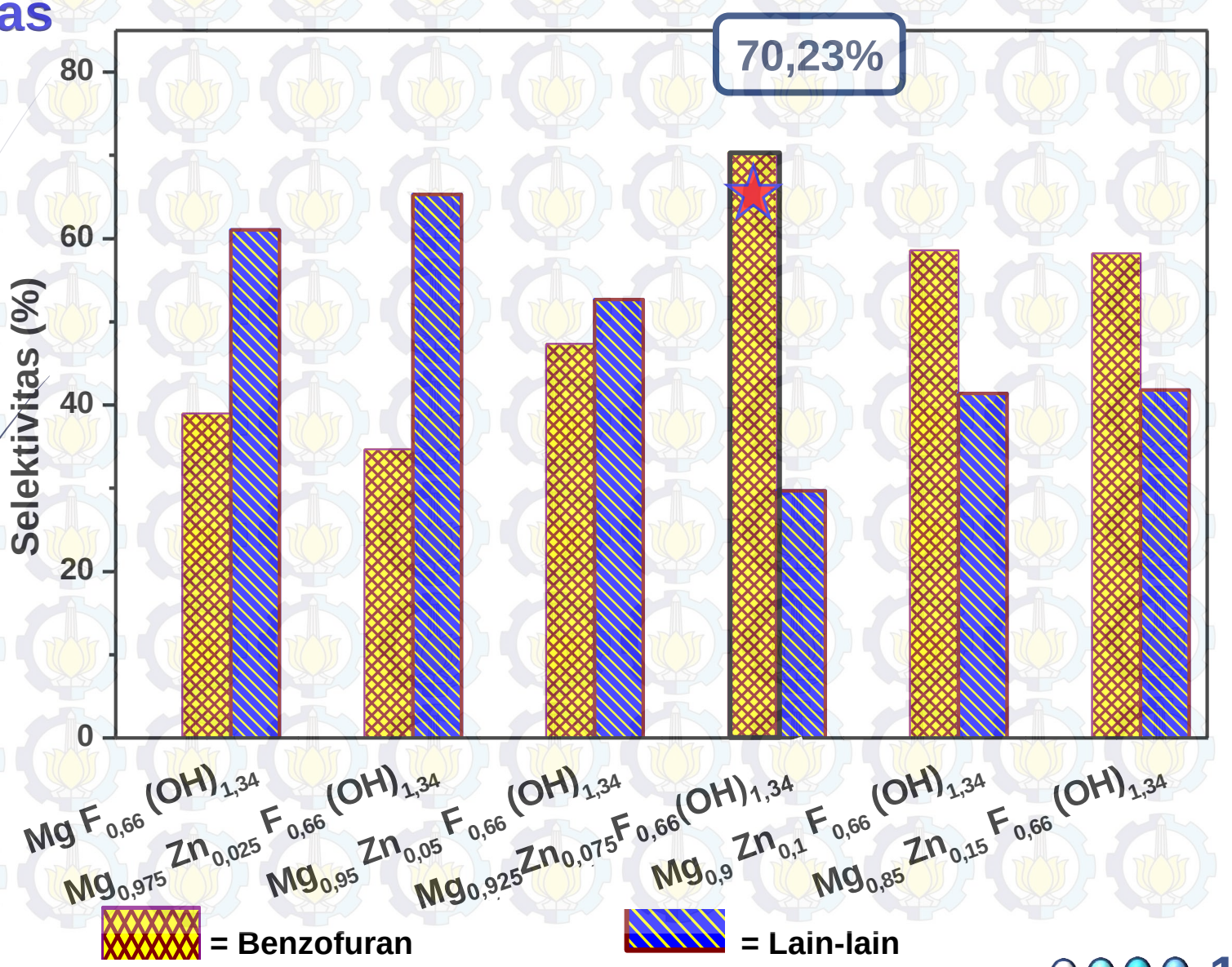
Konversi & *Yield*



= Konversi = Benzofuran = Lain-lain

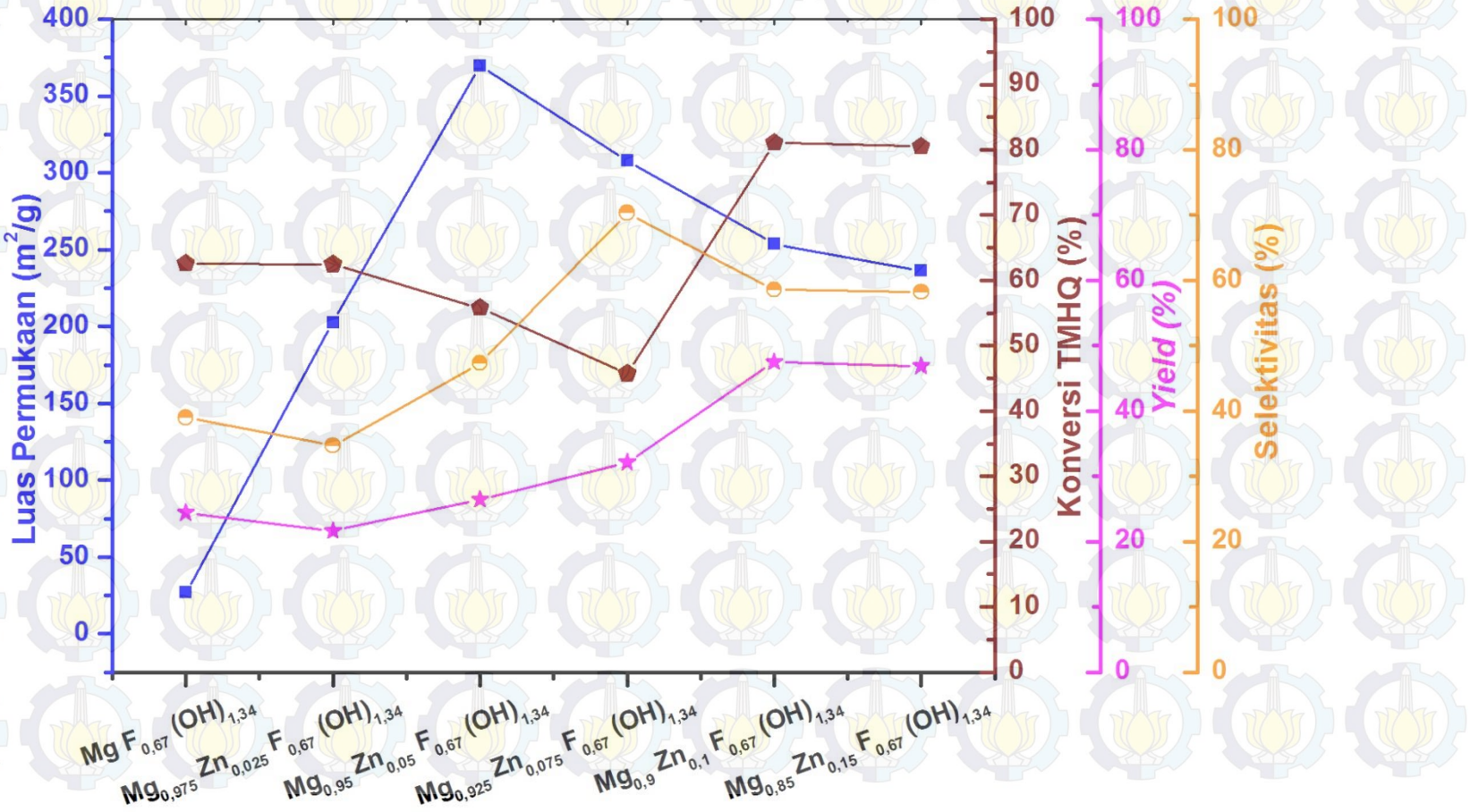
HASIL DAN PEMBAHASAN

Selektivitas

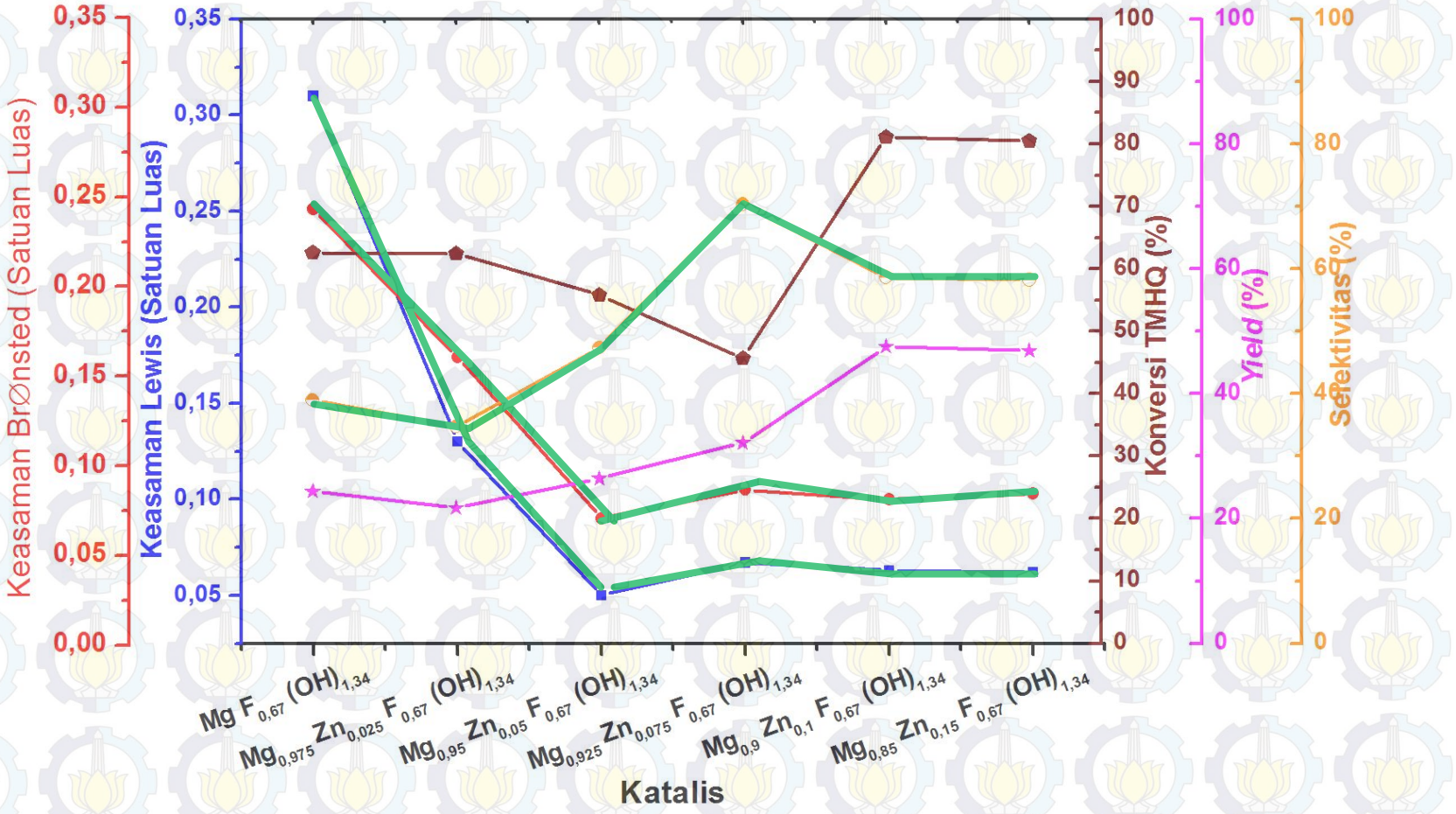


HASIL DAN PEMBAHASAN

Korelasi antara luas permukaan pada katalis dengan konversi TMHQ, *yield* dan selektivitas benzofuran



Korelasi antara keasaman katalis dengan konversi TMHQ, *yield* dan selektivitas benzofuran



4 KESIMPULAN

$Mg_{1-x}Zn_xF_{0,66}(OH)_{1,34}$

X= 0 | X= 0,025 | X= 0,05 | X= 0,075 | X= 0,1 | X= 0,15

Konversi TMHQ	*Yield*	Selektivitas
81,08%	47,53 %	Benzofuran
	Benzofuran	70,32%

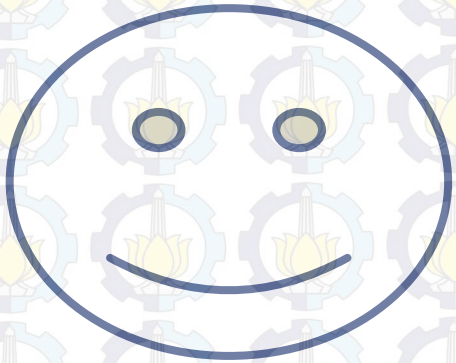
Keasaman Katalis ✓

Luas Permukaan ✗

TERIMA KASIH

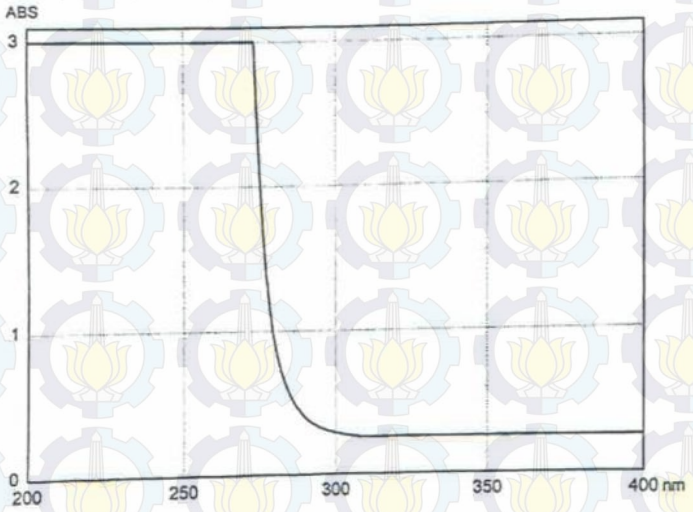
- ✓ Prof. Dr. rer. nat. Irmina Kris Murwani
- ✓ Tim Dosen Penguji
- ✓ Tim Penelitian Katalis Heterogen
- ✓ Semua pihak yang telah mendukung dalam penyusunan Tugas Akhir ini

Terima Kasih



UV1100 Spectrophotometer

Serial NUM: 5210027
ROM Version: 20
Sample Name:
Date:
Operator:



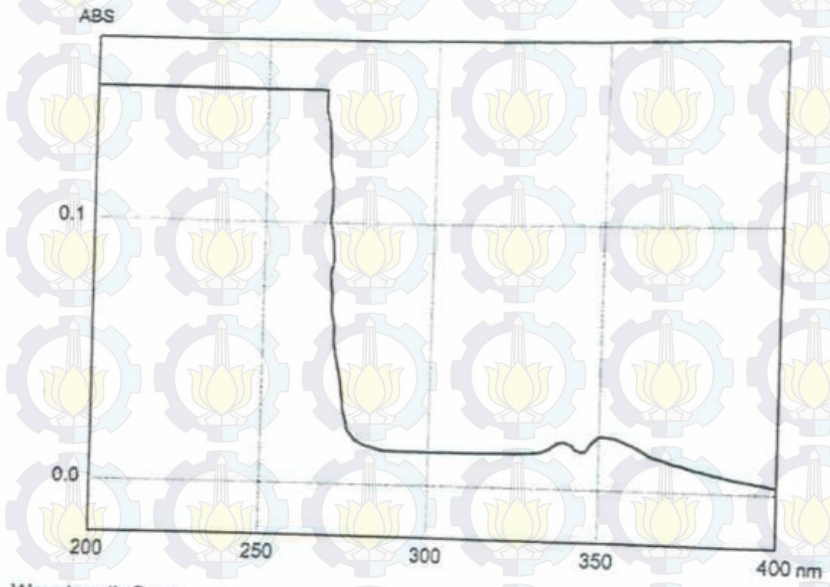
Wavelength Scan

Data Mode:	ABS
Scan Range:	400.0-200.0nm
Slit Width:	4nm
Speed (nm/min) :	200nm/min
Lamp Change Wavelength:	340.0nm
Path Length:	

Peak WL (nm)	ABS	WL (nm)	ABS

UV1100 Spectrophotometer

Serial NUM: 5210027
ROM Version: 20
Sample Name:
Date:
Operator:



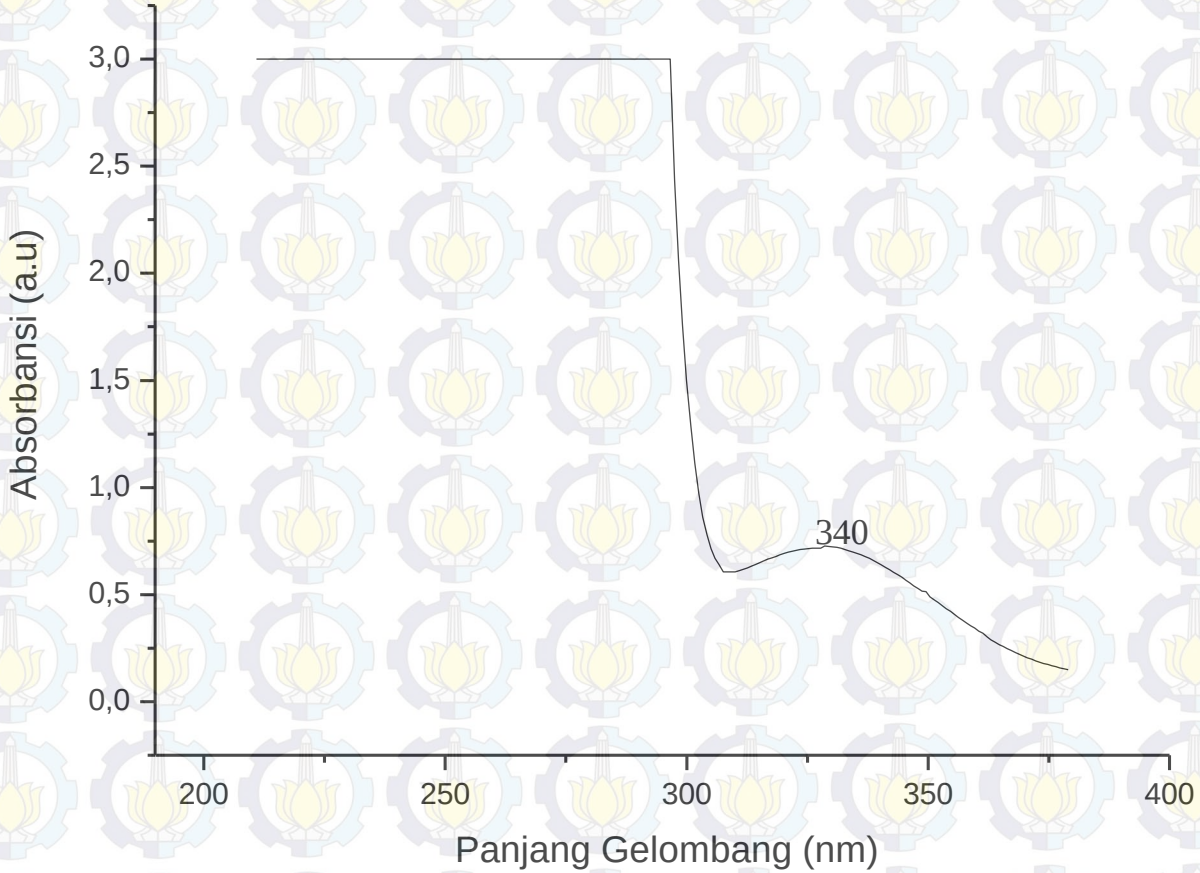
Wavelength Scan

Data Mode:	ABS
Scan Range:	400.0-200.0nm
Slit Width:	4nm
Speed (nm/min) :	200nm/min
Lamp Change Wavelength:	340.0nm
Path Length:	

Peak

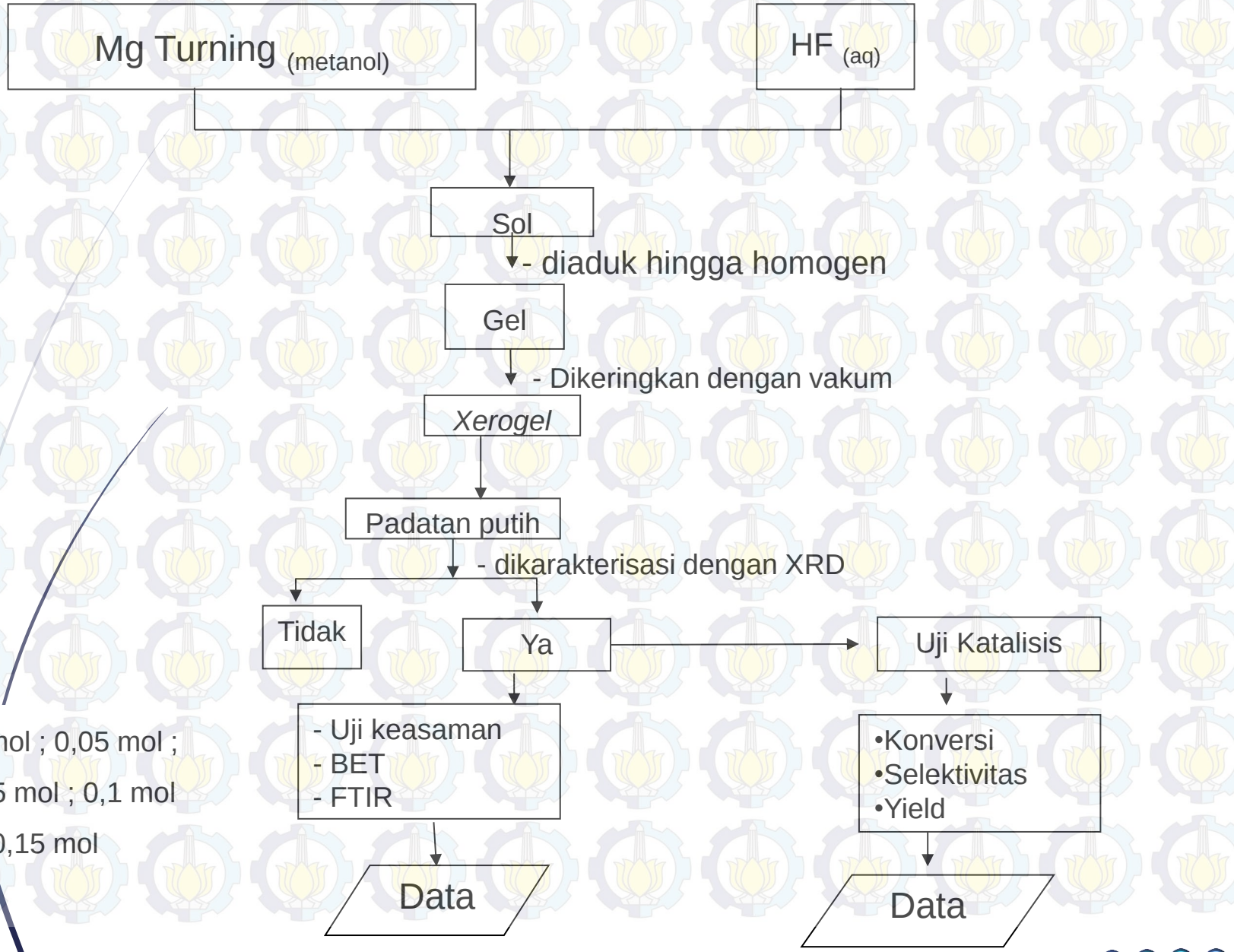
WL (nm)	ABS	WL (nm)	ABS
338.0	0.0165	352.8	0.0253

$Mg_{0,9}Zn_{0,1}F_{0,66}(OH)_{1,34}$



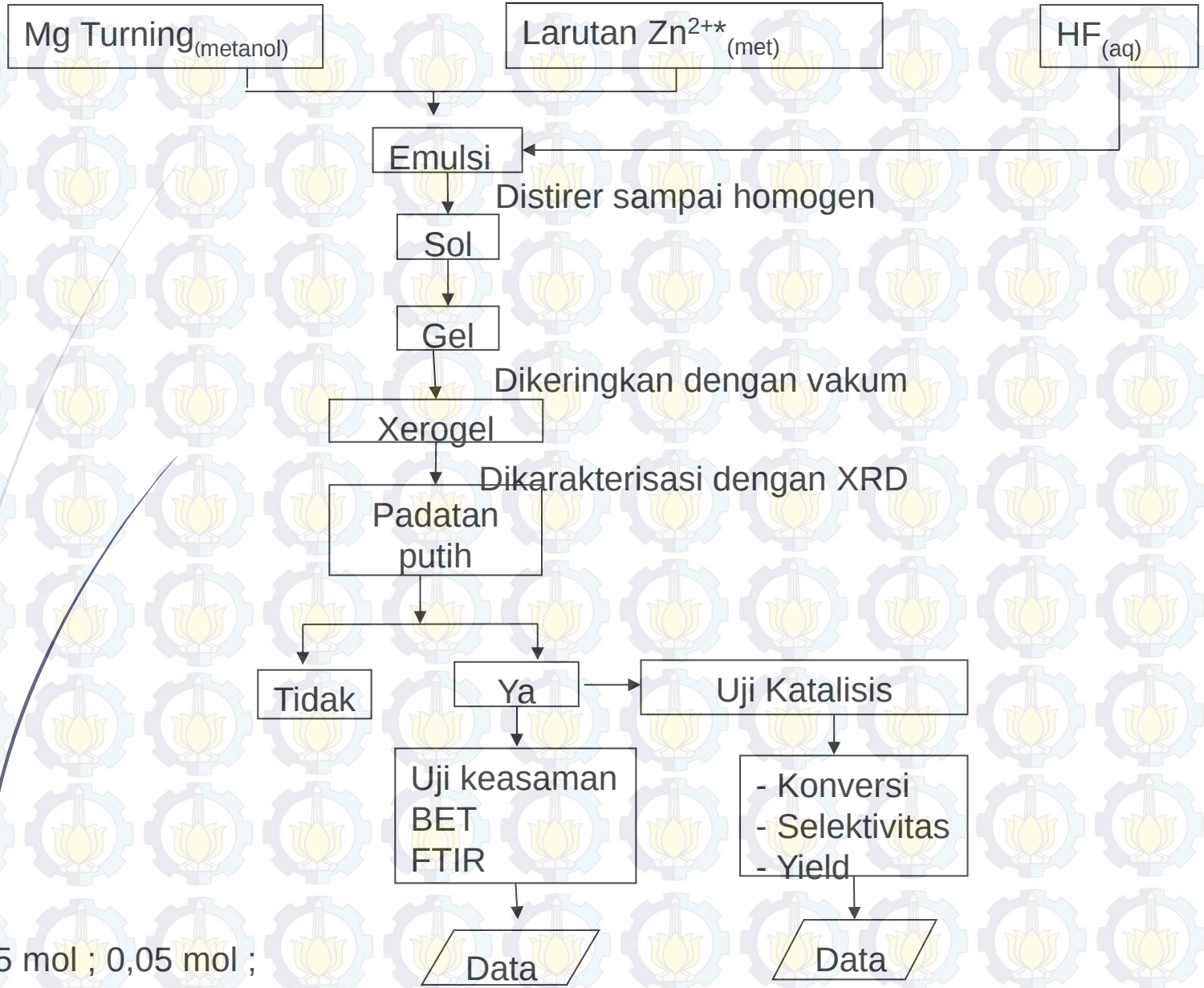
Katalis	Absorbansi
$Mg_{0,975}Zn_{0,025}F_{0,66}(OH)_{1,34}$	0,025
$Mg_{0,95}Zn_{0,05}F_{0,66}(OH)_{1,34}$	0,029
$Mg_{0,925}Zn_{0,075}F_{0,66}(OH)_{1,34}$	0,037
$Mg_{0,9}Zn_{0,1}F_{0,66}(OH)_{1,34}$	0,011
$Mg_{0,85}Zn_{0,15}F_{0,66}(OH)_{1,34}$	0,012

• Sintesis $MgF_{0,66}(OH)_{1,34}$



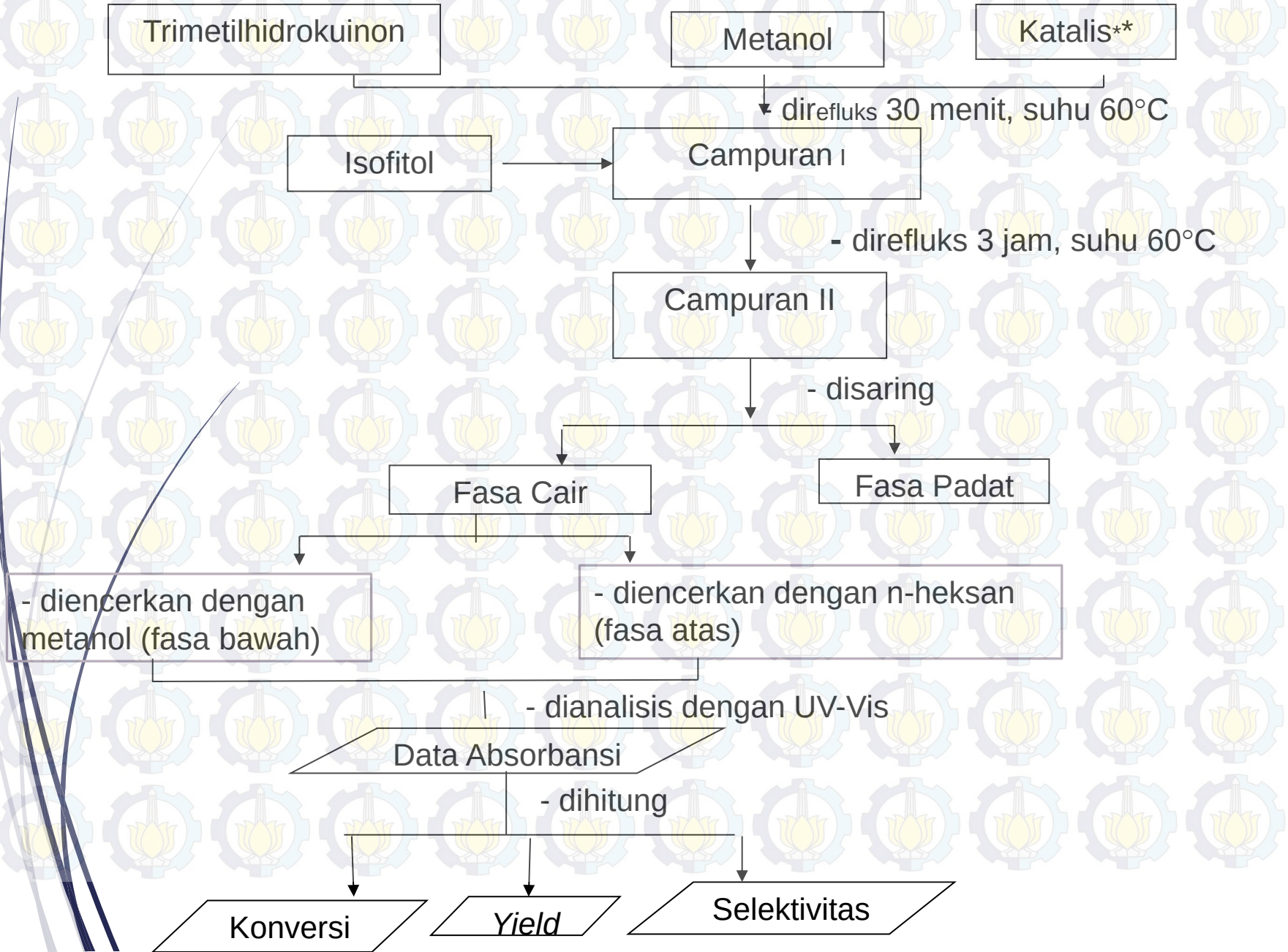
0,025 mol ; 0,05 mol ;
0,075 mol ; 0,1 mol
dan 0,15 mol

Sintesis Katalis $Mg_{1-x}Zn_xF_{0,66}(OH)_{1,34}$



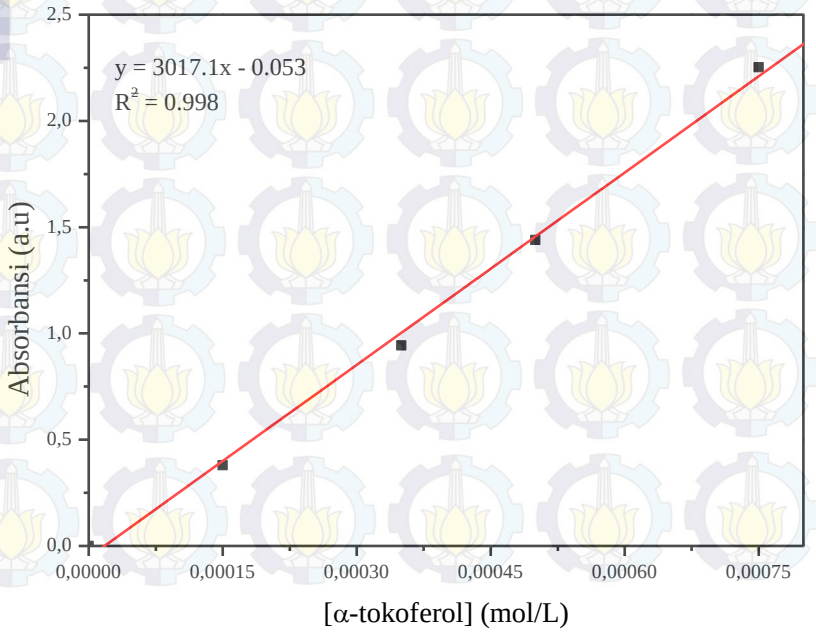
- 0,025 mol ; 0,05 mol ; 0,075 mol ; 0,1 mol dan 0,15 mol

Uji Katalisis pada Reaksi antara Trimetilhidrokuinon dan Isofitol



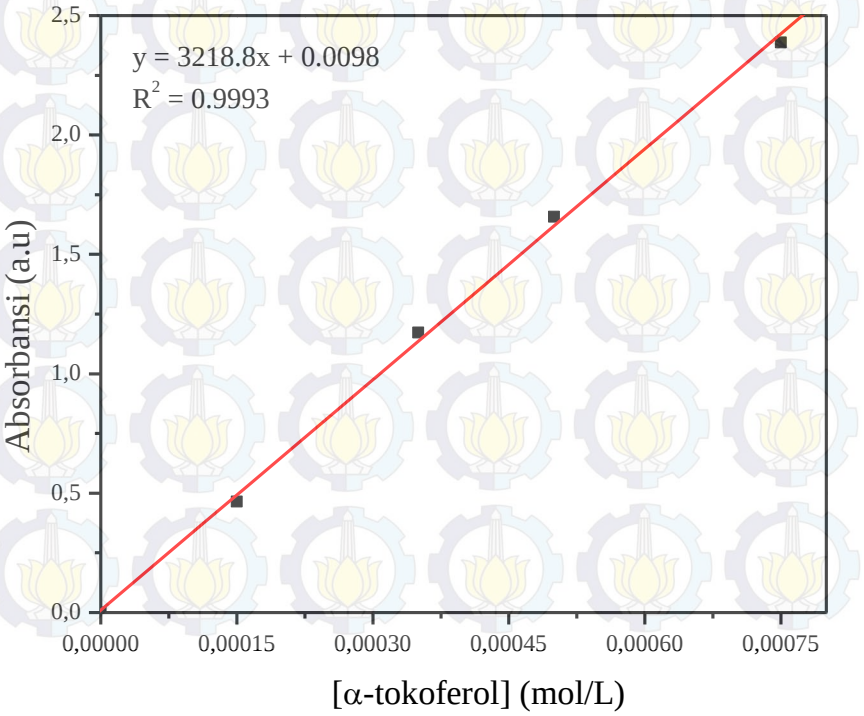
•Kurva Kalibrasi α-tokoferol dalam Metanol

Konsentrasi α-tokoferol (mol/L)	Absorbansi (a.u)
0	0
0,00016	0,00871
0,00048	0,03114
0,0008	0,05776
0,00112	0,078



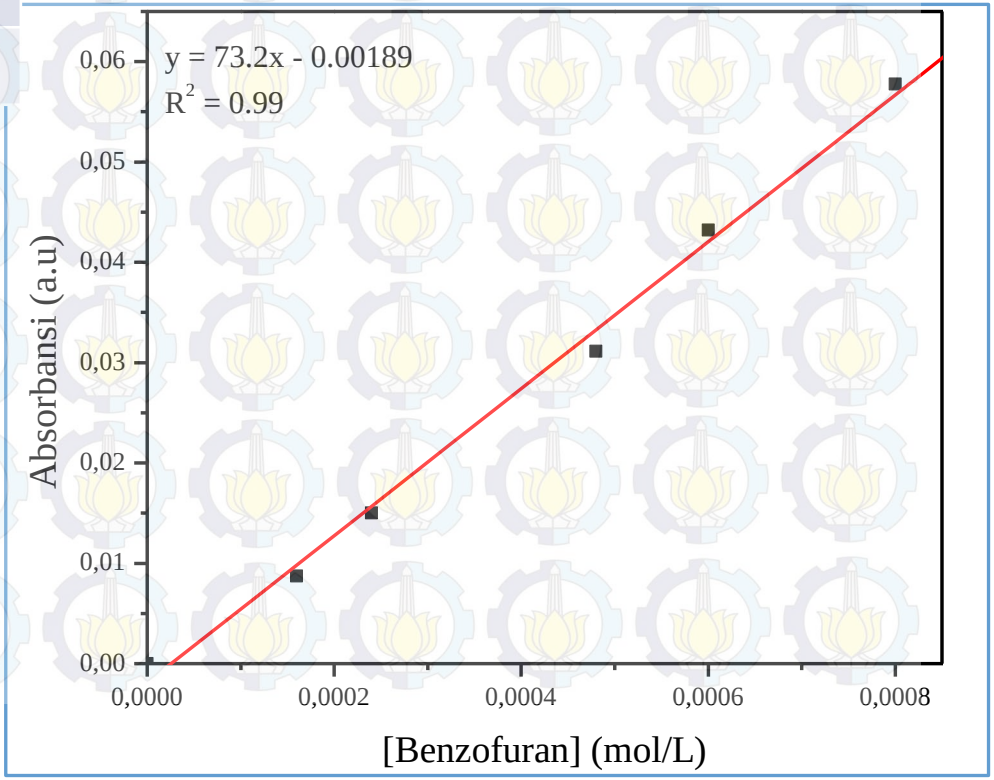
•Kurva Kalibrasi α-tokoferol dalam n-heksana

Konsentrasi α-tokoferol (mol/L)	Absorbansi (a.u)
0	0
0,00015	0,465
0,00035	1,172
0,0005	1,658
0,00075	2,387



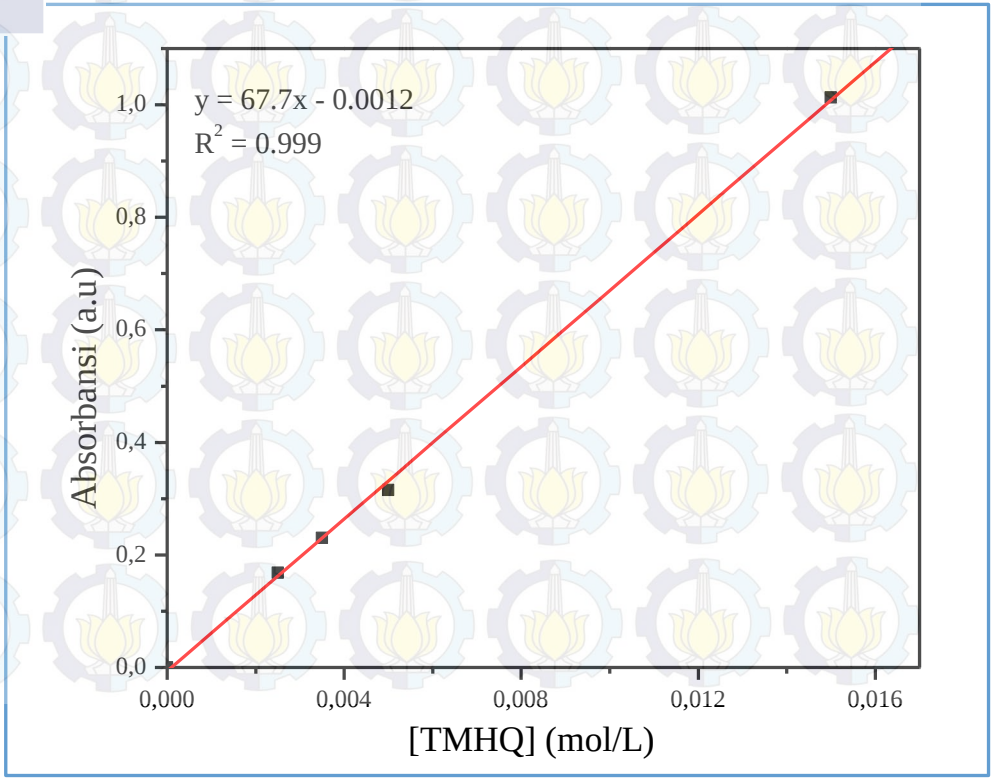
•Kurva Kalibrasi Benzofuran dalam Metanol

Konsentrasi Benzofuran (mol/L)	Absorbansi (a.u)
0	0
0,00016	0,00871
0,00024	0,015
0,00048	0,03114
0,0006	0,0432
0,0008	0,05776



•Kurva Kalibrasi TMHQ dalam Metanol

Konsentrasi TMHQ (mol/L)	Absorbansi (a.u)
0	0
0,0025	0,168
0,0035	0,23
0,005	0,315
0,015	1,013



Pengukuran absorbansi TMHQ pada λ = 352,8 nm adalah 0,025
V = 10 mL = 0,01 L
$TMHQ_{awal}$

Persamaan regresi linear TMHQ yang diperoleh :
y = 67,7x – 0,0012
dimana :
y = A + Bx
dengan, x = Konsentrasi (mol/L)
y = Absorbansi
mol TMHQ sisa = 0,000387 mol/L x 0,01 L x 10
= 0,0000387 mol
= 0,0387 mmol
Konversi atau Aktivitas

Yield dan Selektivitas

Pengukuran absorbansi benzofuran pada λ = 338 nm adalah 0,0142.

V = 10 mL = 0,01 L

Persamaan regresi linear α-tokoferol yang diperoleh :

y = 73,2x – 0,00189

dimana :

y = A + Bx

dengan, x = Konsentrasi (mol/L)

y = Absorbansi

mol benzofuran = 0,000219 mol/L x 0,01 L x 10

= 0,0000219 mol

= 0,0219 mmol

Yield

= 21,89 %

Selektivitas

Sebelum kalsinasi

Setelah kalsinasi

Katalis	Konversi (%)	Yield (%)	
		Benzofuran	Lain-lain
$MgF_{0,67}(OH)_{1,34}$	62,582440	24,389828	38,192612
$Mg_{0,975}Zn_{0,025}F_{0,67}(OH)_{1,34}$	62,443283	21,666088	40,777195
$Mg_{0,95}Zn_{0,05}F_{0,67}(OH)_{1,34}$	55,802965	26,417057	29,385908
$Mg_{0,925}Zn_{0,075}F_{0,67}(OH)_{1,34}$	45,693346	32,132467	13,560878
$Mg_{0,9}Zn_{0,1}F_{0,67}(OH)_{1,34}$	81,079821	47,525754	33,554067
$Mg_{0,85}Zn_{0,15}F_{0,67}(OH)_{1,34}$	80,533304	46,876282	33,657022

Katalis	Selektivitas (%)	
	Benzofuran	Lain-Lain
$MgF_{0,67}(OH)_{1,34}$	38,972319	61,027681
$Mg_{0,975}Zn_{0,025}F_{0,67}(OH)_{1,34}$	34,697227	65,302773
$Mg_{0,95}Zn_{0,05}F_{0,67}(OH)_{1,34}$	47,339881	52,660119
$Mg_{0,925}Zn_{0,075}F_{0,67}(OH)_{1,34}$	70,321984	29,678016
$Mg_{0,9}Zn_{0,1}F_{0,67}(OH)_{1,34}$	58,616007	41,383993
$Mg_{0,85}Zn_{0,15}F_{0,67}(OH)_{1,34}$	58,207325	41,792675

Perhitungan Sintesis $Mg_{1-x}Zn_xF_{0,66}(OH)_{1,34}$

$$(1\text{-}x)\ Mg(OCH_3)_{2\ (met)} + x\ Zn(CH_3COO)_{2\ (met)} + 2HF_{(aq)} + 2\ H_2O$$

$$\downarrow$$

$$Mg_{1-x}Zn_xF_{0,66}(OH)_{1,34} + 1{,}34\ HF + 2x\ CH_3COOH + 2(1\text{-}x)\ CH_3OH$$

- Massa senyawa target = 2 g
  - Menghitung Mr produk

Mg	= 0,975 x 24,38	= 23,4
Zn	= 0,025 x 65,37	= 1,63
F	= 0,66 x 19	= 12,54
OH	= 1,34 x 17	= 22,78
		= 60,35 g/mol

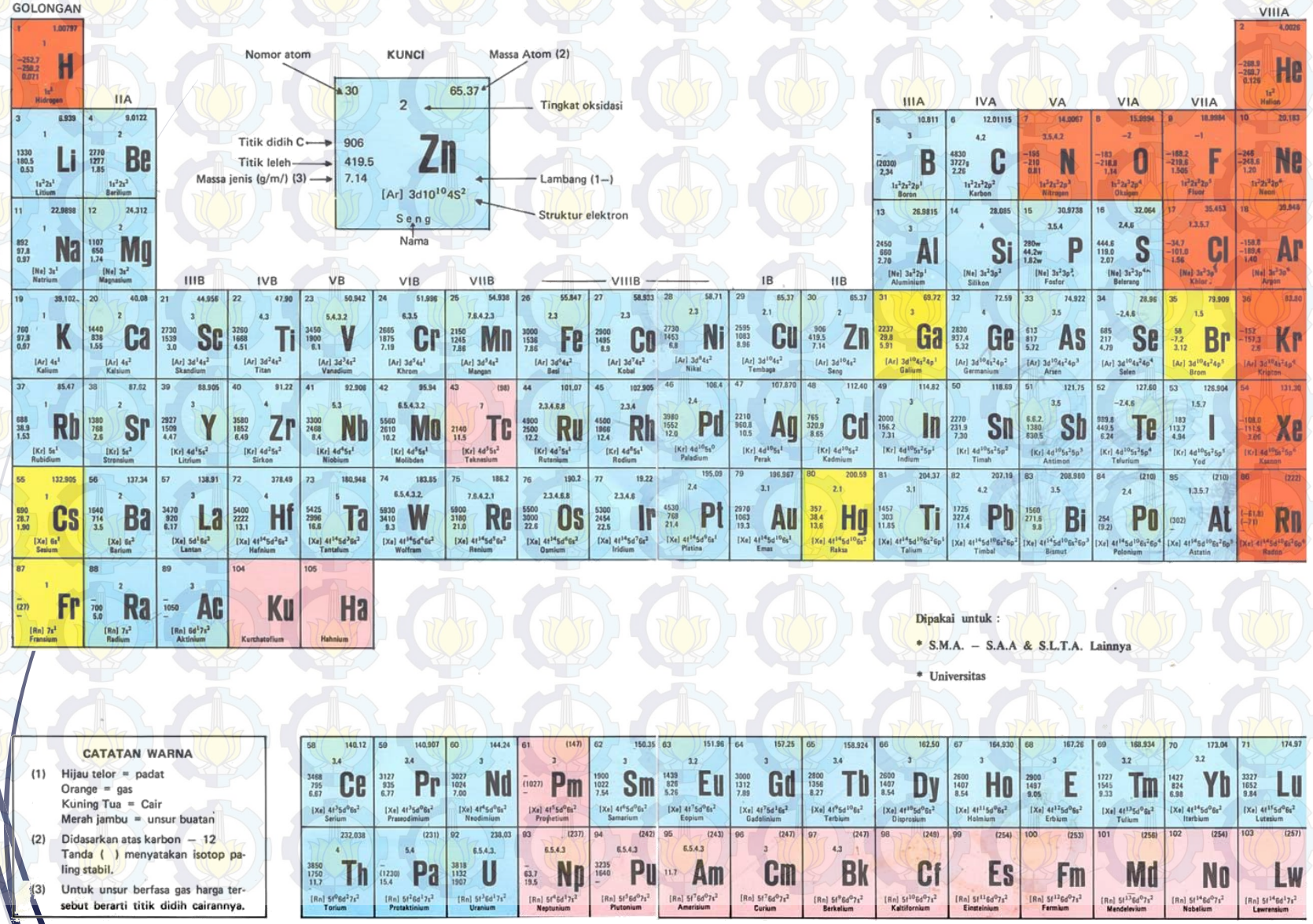
Mol Produk = $\frac{2}{60{,}35}$ = 0,033 mol

Kebutuhan Mg Turning = 0,975 x 0,033 x 24,3 = 0,782 g

Kebutuhan HF = $\frac{0{,}033}{27{,}12}$ x 0,67 x 1000 = 0,82 mL

Kebutuhan Logam Zn = 0,025 x 0,033 x 219,49 = 0,1811

TABEL PERIODIK
UNSUR KIMIA



Dipakai untuk :

* S.M.A. – S.A.A & S.L.T.A. Lainnya
* Universitas

CATATAN WARNA

(1) Hijau telor = padat
Orange = gas
Kuning Tua = Cair
Merah jambu = unsur buatan

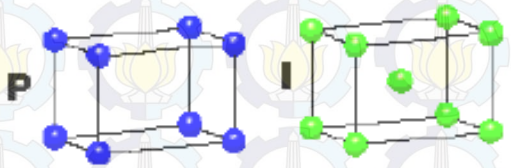
(2) Didasarkan atas karbon – 12
Tanda ( ) menyatakan isotop paling stabil.

(3) Untuk unsur berfasa gas harga tersebut berarti titik didih cairannya.

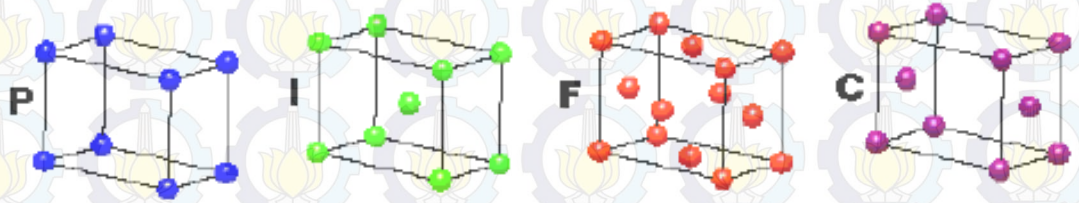
Geometri kristal

CUBIC
$a = b = c$
$\alpha = \beta = \gamma = 90°$

TETRAGONAL
$a = b \neq c$
$\alpha = \beta = \gamma = 90°$

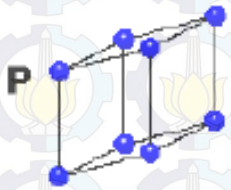


ORTHORHOMBIC
$a \neq b \neq c$
$\alpha = \beta = \gamma = 90°$

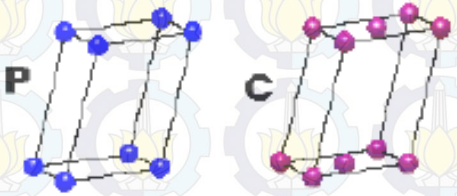


HEXAGONAL
$a = b \neq c$
$\alpha = \beta = 90°$
$\gamma = 120°$

TRIGONAL
$a = b = c$
$\alpha = \beta = \gamma \neq 90°$



MONOCLINIC
$a \neq b \neq c$
$\alpha = \gamma = 90°$
$\beta \neq 120°$



TRICLINIC
$a \neq b \neq c$
$\alpha \neq \beta \neq \gamma \neq 90°$

4 Types of Unit Cell
P = Primitive
I = Body-Centred
F = Face-Centred
C = Side-Centred
+
7 Crystal Classes
→ 14 Bravais Lattices

	Mixed powders	*Coprecipitation*	*Sol–gel*	*Hydrothermal*	*Spray and freeze drying*
State of development	Commercial	Commercial	Commercial; research and development	Demonstration	Demonstration
Size of particle (nm)	>1000	>10	>10	>100	>10
Homogeneity	Poor	Good	Very good	Very good	Very good
Purity	Poor	Very good	Excellent	Very good	Excellent
Temperature of calcination (°C)	>1000	500–1000	500–1000	80–374	>150
Agglomeration	Moderate	High	Moderate	Low	Low
Costs	Low to moderate	Moderate	Moderate to high	Moderate	Moderate to high